



Aalborg Universitet

AALBORG UNIVERSITY  
DENMARK

## Formation of a nano-crystalline layer on the surface of glass fibres

Yue, Yuanzheng; Korsgaard, Martin; Kirkegaard, Lise; Heide, Gerhard

*Publication date:*  
2005

*Document Version*  
Accepted author manuscript, peer reviewed version

[Link to publication from Aalborg University](#)

*Citation for published version (APA):*

Yue, Y., Korsgaard, M., Kirkegaard, L., & Heide, G. (2005). *Formation of a nano-crystalline layer on the surface of glass fibres*. Abstract from International Conference on Materials for Advanced Technology, Singapore.

### General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal -

### Take down policy

If you believe that this document breaches copyright please contact us at [vbn@aub.aau.dk](mailto:vbn@aub.aau.dk) providing details, and we will remove access to the work immediately and investigate your claim.

## Formation of a nano-crystalline layer on the surface of glass fibres

Yuanzheng Yue\*, Martin Korsgaard & Lise Kirkegaard  
Section of Chemistry, Aalborg University, Denmark

Gerhard Heide  
Department of Non-Metallic Materials, Clausthal University of Technology, Germany

The Fe-Mg-Ca-aluminosilicate glass (FMCAS glass) starts to crystallize at an onset temperature ( $T_{\text{onset}}$ ) well above its glass transition temperature ( $T_g$ ), when it is upscanned at 20 K/min in a calorimeter. Such crystallization is here referred to as high temperature (HT) crystallization. The details (e.g. extent, rate and  $T_{\text{onset}}$ ) of the HT crystallization depend on chemical composition, heating atmosphere, and heating rate, of the glass. This is already known in literature. What is less well known is that the FMCAS glass also crystallizes around  $T_g$  far below the  $T_{\text{onset}}$  of the HT crystallization, if the glass contains certain amount of iron, and has sufficiently high  $\text{Fe}^{2+}/\text{Fe}^{3+}$  ratio, and is heat-treated in oxidizing atmosphere. This crystallization process is featured by the formation of nano-crystals (e.g. periclase) on the glass surface, which is driven by two simultaneous processes: oxidation of  $\text{Fe}^{2+}$  and outward cationic diffusion. Such crystallization is here referred to as low temperature (LT) crystallization. The LT crystallization is not detectable by calorimetry. Therefore, other techniques such as secondary neutral mass spectrometry, atomic force microscopy and X-ray diffraction have been employed to identify the type, the structure, and the size of crystals formed upon LT heat-treatment. Differential scanning calorimetry has been used to measure thermal response to the HT crystallization and oxidation of  $\text{Fe}^{2+}$ . Mössbauer spectroscopy has been used to determine the  $\text{Fe}^{2+}/\text{Fe}^{3+}$  ratio. Here we present results of LT crystallization of the FMCAS glass fibres, and describe the mechanisms of both LT and HT crystallization processes. The results show that the amount of the nano-crystals formed at lower temperatures has strong impact on the relative ratio of different crystalline phases occurring during the subsequent HT heating, and on the extent of HT crystallization. The nano-crystalline layer influences the physical and chemical properties of the FMCAS glass fibres.